SPACE PROCESSING OF ELECTRONIC CRYSTALS

E. C. Henry and L. R. McCreight

GENERAL ELECTRIC COMPANY
PHILADELPHIA, PENNSYLVANIA

ABSTRACT

A method for growing various high temperature oxidic crystals is described. The crystals are precipitated and grown from a glass solvent in which the desired crystal constituents have been previously dissolved. Typically, the desired crystals have a density of 5-8 grams/cc, and they settle out rapidly, due to gravity, in the much less dense molten glass matrix.

It is proposed to conduct a space experiment on this process of growing crystals as a means of overcoming the gravitational effects, and equally importantly, at the same time conducting the experiment in a manner to obtain crystals with fewer vacancies and dislocations as a consequence of the near absence of convection that zero-g should provide. This latter factor is expected to provide a means for achieving crystals with much higher perfection and improved electronic properties.

Although several compositions of crystals have been grown, the recommended choice for a space experiment is potassium sodium niobate because it has not been previously available as adequate single crystals and has high technological value.

A. INTRODUCTION

The possibilities of growing crystals in space, in an environment essentially free of the influence of gravity and having other peripheral advantages, are quite challenging. One is excited particularly by the potentialities of growing larger, more defect-free crystals of high value materials, with concomitant improvements in properties, while at the same time gaining important new insight into the mechanisms of crystal growth.

In planning an experiment to demonstrate effectively the advantages to be gained on processing crystals in space, it is necessary to select both an appropriate method of crystal growth and an appropriate composition to be grown. At the outset, one is impressed by the fact that, for operations in space, growth from a solution is one of the most appropriate general methods of crystal preparation. It can also be stated emphatically that electronic ceramic (oxidic) compositions are prime candidates for such crystal growth.

Crystals of electronic ceramic compositions are recommended for preparation in space, for the following reasons:

1. They have high dollar value per unit of volume or weight.

2. They have high density (up to 8 gm/cc) which makes it difficult to grow them under the influence of gravity. (They tend to settle and pack in the bottom of the crucible in which they are grown.)

3. Electronic ceramics are at the heart of the technology of many areas in the field of electronics: communication equipment, computers, radar and sonar equipment, infrared sensors, lasers, and the miniaturization of electronic circuits. They perform unique functions essential to the operation of many very complex measuring, counting, sensing or controlling devices, instruments, and systems.

4. Many electronic ceramics are used in polycrystalline form because they are not produced at all as single crytals or cannot be supplied in adequate size or perfection. The availability of satisfactory single crystals of these compositions could result in significant improvements in properties -- increased efficiency or sharper response, for example. Only slight improvements in performance over the best that is presently available would be technologically important in many instances. Improved materials are needed today to permit further advances in the state-of-the-art.

5. Oxidic compositions are disproportionately important among electronic single crystal materials. The results of discussions with growers and users in the field indicate that silicon, preferably but not exclusively made by float zone refining, dominates the high value single crystal field and accounts for an estimated 90% of the single crystal materials used in electronics. Other single-crystal materials such as germanium and gallium arsenide then account for much of the remaining usage. Nevertheless, a large number of compositions and crystals are being grown for specialized functions that cannot be performed by the semiconductors. Because these titanates, niobates, etc. commonly are used as relatively small pieces, comprising the whole or parts of electronic devices or systems, it seems reasonable to expect that there could be a ready market for superior space-produced crystals of this type of material.

6. As a bonus, the electronic properties of these oxidic materials are very sensitive to purity, crystal structure, and crystal perfection. As a consequence, measurements of electronic performance can serve as a powerful aid in evaluating the quality of the crystals produced. Specifically, such measurements provide a most effective means of directly comparing the quality of crystals prepared in space with those prepared under normal gravity.

B. SELECTION OF COMPOSITIONS OF CRYSTALS TO BE GROWN

Numerous compositions of oxidic electronic materials are candidates for space crystal growth. These compositions include (1) those predominantly showing ferroelectric and piezoelectric properties such as barium titanate, potassium sodium niobate, lead metaniobate, lead metatantalate, and the lead zirconate titanates, (2) those having strong magnetic characteristics, including the magnetic spinels such as lithium, nickel, cobalt, etc. ferrites and the magnetic garnets YIG (yttrium iron garnet) and YAG (yttrium aluminum garnet); (3) electro-optical materials such as lithium niobate and barium sodium niobate; (4) the dielectrics, characterized by high electrical resistivity and high energy-storage capabilities, and (5) single-crystal low-attenuation acoustic materials.

1. Potassium-Sodium Niobate, PSN, $K_{0.5}Na_{0.5}NbO_3$, Recommended for Preparation in Space

Among the electronic compositions listed, potassium sodium niobate (PSN) is one of the leading candidates for crystal-growth. This material is a solid solution of potassium niobate and sodium niobate, but it has more desirable electronic properties than either ingredient alone. It is difficult to obtain dense ceramic pieces of this material by conventional sintering, and special procedures of hot-pressing and pressure-sintering must be employed. Single crystals of this composition are not readily pulled from the melt, but growth from a molten solution appears quite promising.

Potassium sodium niobate has high electromechanical coupling efficiency, low dielectric constant, and high acoustic velocity. These properties combine to make the material unusually desirable for use as a transducer for high frequency operations: for filters, resonators, frequency control units, and the like. Small wafers of such material can be prepared very thin, capable of resonating at high frequency, without having to develop unwanted high capacitance locally in the circuit. The high acoustic velocity further permits operation at a higher frequency for a given thickness of material. Polarized transducers of potassium sodium niobate, 0.015 x 0.015 x 0.75 inch in size, for example, sliced from a larger crystal, should give superior performance in various ultrasonic and dispersive delay lines, some of which cost up to $5000

each. Highly sophisticated delay lines are employed in the electronics of the Sentinal system, while others are widely used in European color television circuits.

Potassium sodium niobate has other technical advantages. The material can be used effectively over a wide temperature range. There is very little change in dielectric constant, for example, from $-10^{\circ}C$ to $+225^{\circ}C$, at which temperature the material changes from one ferroelectric crystal structure to another. PSN does not lose its ferroelectricity until above $400^{\circ}C$, whereas barium titanate ceases to be ferroelectric when heated only to $120^{\circ}C$.

The properties of $K_{0.5}Na_{0.5}NbO_3$ reported by Jaeger and Egerton[1] are of interest because of the improvements found on hot pressing this material as compared with air firing (conventional sintering), as shown in Table I. Enhanced piezoelectric coefficients were noted, and the radial electromechanical coupling coefficient was increased from 32 to approximately 48%. The authors felt that the new material was desirable for electromechanical transducers, particularly in the 10 to 20 MHz range. Still further improvements should be found when single crystals are measured.

The properties of selected transducer materials are compared with those of PSN in Table II. Note that PSN is about equally efficient (has good coupling coefficients) in both shear mode and longitudinal mode operations. Its coupling coefficients are higher than that of barium sodium niobate in shear and greater than lithium niobate longitudinally. Although PZT has high coupling coefficients, its velocity of sound is too low and its dielectric constant is too high for it to be used as a high-frequency transducer. It should be noted, also, that the values for PSN and PZT are for polycrystalline (ceramic) materials whereas the other materials are in single crystal form.

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(1) R. E. Jaeger and L. Egerton, "Hot Pressing of Potassium-Sodium Niobates", J.Am.Ceram.Soc. 45 (5), 209-13 (1962).

TABLE I

Characteristics of $K_{0.5}Na_{0.5}NbO_3$ Ceramics at Room Temperature

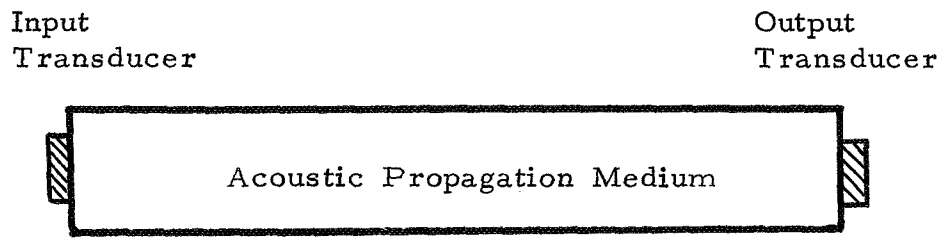
	Air Fired	Hot Pressed
Dielectric constant (100 KHz)	290	420
Electrical Q (100 KHz)	~50	~70
Specific resistivity (ohm-cm)	$\sim 10^{12}$	$\sim 10^{12}$
Density	4.25	4.46
Frequency constant (cycles · meters)	1650	1700
Mechanical Q, radial	130	240
Young's modulus (newtons/m^2)	1.04×10^{11}	1.5×10^{11}
Piezoelectric constants		
d_{31} (coulombs/newton)	32×10^{-12}	49×10^{-12}
g_{31} (meter-volts/newton)	12.6×10^{-3}	13.1×10^{-3}
d_{33} (coulombs/newton)	80×10^{-12}*	160×10^{-12}
g_{33} (meter-volts/newton)	31.5×10^{-3}*	43×10^{-3}*
Electromechanical coupling coefficients		
k_r (radial)	0.36	0.45
k_{31}	0.22	0.27
k_{33}	0.51*	0.53

* Estimated values

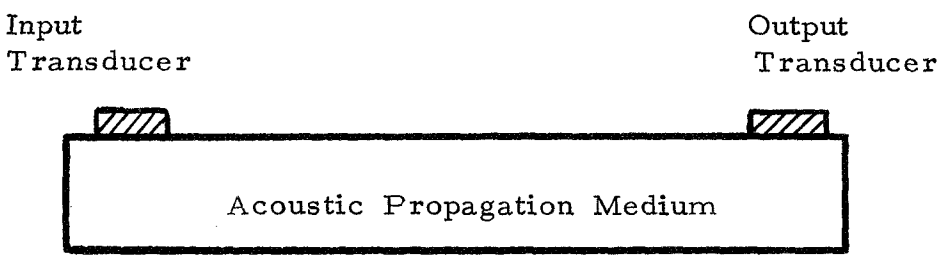
Delay lines such as might use PSN transducers are shown schematically in Figure I. An electrical signal passes along a wire at about the speed of light. A sound signal travels at different rates in different materials, but commonly of the order of 1/100,000 the speed of light. The electrical signal to be delayed is fed into a transducer, where it is converted to an acoustic wave. The acoustic energy passes through the sonic transmission medium to the second transducer in which it is converted again into an electrical signal. The acoustic energy may be transmitted through the bulk of the medium or along the surface, depending on the performance characteristics required. A sonic delay line in which two transducers are separated by four inches of glass, for example, can delay an electrical signal by forty microseconds, as long as it would take the electrical signal itself to pass along nearly forty thousand feet of wire. This is only one of the applications of PSN in electronics.

C. CRYSTAL GROWTH FROM MOLTEN SOLUTION

When growing crystals of electronic ceramic compositions from a molten solution, as opposed to growing from a straight melt, most investigators to-date have used molten salts and simple oxides as the solvents, as shown in Table III. Potassium fluoride, bismuth oxide, lead oxide, boron oxide and barium chloride are among the materials so used. For space processing, however, various more complex solvents, chiefly silicate glasses, might be more effective. There are a number of reasons why this should be so. Molten silicate glasses should have much higher viscosity than molten salts, and have less tendency to wet and creep along the walls of the platinum crucible. For both of these reasons, the melt should tend to remain in the container better than otherwise. The glasses would be more stable, chemically, having lower volatility and less tendency to change in composition during the relatively long time allotted to the crystallization procedures. Silicate glass melts show a slow change of viscosity with change in temperature, in comparison with the abrupt changes experienced with molten salts. The groundmass, being glassy and viscous, will inhibit the formation of spontaneous nuclei during cooling and will not, itself, crystallize around the already-grown crystals. One must not overlook the obvious advantage of all the molten solvent systems, that one can prepare a crystal from solution at a convenient temperature well below the melting point of that composition of crystal.



A. Bulk Wave Configuration



B. Surface Wave Configuration

Figure 1. Arrangements of Transducers and Acoustic Transmission Material in a Sonic Delay Line.

TABLE II

PHYSICAL CONSTANTS FOR SELECTED TRANSDUCER MATERIALS

	(Shear Mode Operation)			(Longitudinal Mode Operation)			
Material	$V_S \times 10^5$ cm/sec	Impedance ρc	Coupling k_s	$V_L \times 10^5$ cm/sec	Impedence ρc	Coupling k_t	Dielectric Constant
X cut Quartz	3.8	10	0.137	5.7	15.2	0.10	4.58
Y cut Quartz	5.5	13	0.11				4.58
AC cut Quartz	3.2	10	0.13				4.58
Lithium Niobate	3.7	!7.8	0.68	7.2	34	0.19	30-80
Z Barium Sodium Niobate	3.58			6.53	32.6	0.57	30
X-Y Barium Sodium Niobate	3.58 3.37		0.2 0.2	7.27 6.98			30
PZT PSN	2.3 3.0	16 13.5	0.7 0.64	4.4 6.2	31 28	0.7 0.6	1200 375

TABLE III

CRYSTALS GROWN FROM MOLTEN SALTS[2]

Composition	Flux	Temp. Range (°C)	Rate of Cooling (°/hr)	Crystals Leached from Matrix with
$BaTiO_3$	KF	1200 - 850	1 - 5	Hot water
$Bi_4Ti_3O_{12}$	Bi_2O_3	1200 - 800	2 - 5	Strong mineral acids
$KNbO_3$	KF/KCl	1100 - 800	1 - 10	Hot water
YIG ($Y_3Fe_5O_{12}$)	PbO	1350 - 930	1 - 5	Hot 25% NHO_3
YIG	$PbO-PbF_2$	1260 - 950	0.5 - 5	Hot 25% HNO_3 50% HAc
YIG	$BaO-B_2O_3$	1260 - 900	1	Hot 25% HNO_3
YAG	$PbO-B_2O_3$	1250 - 950	1	Hot 25% HNO_3
RFe_2O_4	PbO	1300 - 800	10	Hot 25% HNO_3
$NiF3_2O_4$	$Na_2B_4O_7 \cdot 10H_2O$	1330 - 1250	2	Hot HNO_3

(2) J. J. Gilman, The Art and Science of Growing Crystals, (1963) John Wiley and Sons, Inc., New York, pp. 268-269.

Table IV lists some of the crystal-glass systems that have been investigated at the Space Sciences Laboratory. PSN can be grown readily from potassium sodium silicate glasses. Other electronic oxidic compositions can be grown from similar glasses.

1. Advance Preparation of Space Experiment Materials

There is an outstanding advantage of using a molten glass rather than a molten salt as the solvent. Because of the unique properties of glass, described above, it is possible to prepare the space experiment in advance. Carrying out this operation prior to the space flight saves time, conserves spacecraft power, and helps assure process reproducibility. One is able to dissolve the electronic ceramic constituents in the glass in the earth environment. The molten solution can be held for any desired length of time, 24 hours for instance if necessary, to homogenize the materials. Extraneous nuclei are dissolved and bubbles are removed as the glass is "fined". The melt can be examined, to assure that all of the charge has been taken into solution. The melt is then cooled rapidly to form a glass of almost optical quality, which again is inspected. During space operations it is necessary only to remelt the glass, reach a satisfactory temperature uniformity in the crucible, and begin the crystal-growing operation.

2. Slow Cooling of Melt Essential

When growing crystals of oxidic materials from a flux, it is necessary to cool the solution slowly in order to produce crystals of good quality. Experts have recommended cooling rates as low as $1/2^{\circ}C$ per hour, which might be impractical in short space flights. Rates of 1° and 2° per hour are commonly reported to yield good results, while 10° per hour appears to be a maximum. We have chosen to investigate 4° per hour, as a practical compromise. It is planned to investigate the feasibility of adjusting the concentrations of the ingredients, that is, the degree of saturation, so that crystallization will take place over a range of approximately $200^{\circ}C$ (50 hours at $4^{\circ}C$ per hour).

TABLE IV INVESTIGATION OF GROWTH OF CRYSTALS OF ELECTRONIC COMPOSITIONS AT GE/SPACE SCIENCES LABORATORY

Electronic Material Incorporated	Composition Of Host Glass	Weight Ratio Electronic Mat'l to Host Glass	Estimated Densities	
			Crystal Phase	Glass Phase
A. Compositions Proved Capable of Being Quenched to Homogeneous Glasses				
$BaTiO_3$	$BaAl_2Si_2O_8$	70-30	6.0	3.3
$BaTiO_3$	$BaO \cdot 2B_2O_3$	70-30	6.0	2.8
$K_{.5}Na_{.5}NbO_3$	$KNaO \cdot 3SiO_2$	80-20	4.6	2.4
Ni, Fe, Co Ferrite	$MnO-Al_2O_3-SiO_2$	---	4.3	---
Ni, Fe, Co Ferrite	$MnO-Al_2O_3-SiO_2$ (33%) $PbO \cdot 0.67SiO_2$ (67%)		4.3	---
$Pb(Zr_{0.53}Ti_{0.47})O_3$	$PbO \cdot 0.67SiO_2$	33-67	7.6	6.9
B. Additional Compositions Under Investigation				
$(BaSr)TiO_3$	$(BaSr)O \cdot 2B_2O_3$	70-30	5.4	2.7
$PbNb_2O_6$	$PbO \cdot 0.03Al_2O_3 \cdot 1.94SiO_2$	50-50	5.8	4.7

D. INVESTIGATION OF CRYSTAL-GROWTH PARAMETERS

Once one has chosen a method of crystal growth and has selected the composition of crystal to be grown, it is necessary to determine the conditions under which good crystals can be grown and recovered. Under some combinations of conditions no crystals will be formed at all; under other conditions a multitude of crystals may be formed, although of poor quality. In any event, it is important to be able to recover the crystals after they have been produced. If the crystals are formed at an elevated temperature in a molten solution and left in place as the liquid cools and is solidified, recovery is best accomplished by leaching away the groundmass, thus exposing the crystals. The leaching agent should be capable of dissolving the groundmass selectively without attacking the primary crystals.

Following are key variables which we have examined with a view to determining their influence in the selected growth system:

1. Composition of solvent (silicate) phase
2. Solubility of solvent and crystal phases
3. Concentration of constituents of the crystal phase incorporated in the solution
4. Temperature at which spontaneous crystallization begins
5. Temperature at which seed is introduced
6. Characteristics of grown crystals

By investigating these variables, one at a time, we are zeroing in on the growth procedure to be recommended for a space experiment.

1. Composition of Solvent Phase

Studies were made of the growth of PSN crystals from solutions of K_2O, Na_2O and Nb_2O_5 in melts of the following silicates. Note, the ratio of K_2O to Na_2O in the solvent is the same as that in PSN. This serves to provide a large common-ion effect.

$KNaO \cdot 3SiO_2$
$KNaO \cdot 2SiO_2$
$KNaO \cdot 1.5SiO_2$
$KNaO \cdot SiO_2$

X-ray diffraction measurements indicate that PSN appears spontaneously as the first crystalline phase from each of these silicates. Within the range studied, the ratio of KNaO to SiO_2 does not seem to influence the nature of the primary phase. The melting points of equivalent solutions were found to decrease with decreasing SiO_2 contact, as did also the lowest temperatures at which the melts could be poured from the platinum crucible.

2. Solubility of Glassy and Crystal Phases

The solubility of the above silicates increased with decreasing SiO_2, that is, the $KNaO \cdot 3SiO_2$ was largely insoluble in hot water while the $KNaO \cdot SiO_2$ glass dissolved almost completely. Crystals of PSN prepared separately were held in warm water for three days; no decrease in weight was observed. It appears, therefore, that water should be a satisfactory leaching agent for these compositions.

3. Effects of Concentration of the Solution

Batches of K_2CO_3, Na_2CO_3, Nb_2O_5 and SiO_2 have been weighed out and melted, representing not only variations in the ratio of KNaO to SiO_2 as reported above, but also variations in the weight percent PSN in the silicate glass. The largest amount of PSN successfully incorporated in the $KNaO \cdot 3SiO_2$ glass was 85 per cent, that is, 85 parts of PSN were dissolved in 15 parts silicate. For the recent crystal-growth studies, the most common concentration has been 60 weight per cent PSN and 40 weight per cent silicate solvent. Qualitative evidence has indicated, however, that the solutions containing 60 per cent PSN tend to become saturated too soon, and lower concentrations (40 and 20 per cent) are being investigated.

4. Temperature of Spontaneous Crystallization

Various melts were made up, stabilized, and allowed to cool at 20° to 50°C per hour. Drops of glass of each melt were poured at about 50° intervals, annealed at 300°C, and then examined with a binocular microscope. The temperatures at which the first crystals (small cubes) were seen was noted, as well as the number and size of crystals appearing in later (cooler) drops. A regular relationship was seen between the composition of the solvent phase and the concentration of PSN constituents, as shown in Table V.

TABLE V

CRYSTALLIZATION CHARACTERISTICS OF VARIOUS MELTS OF PSN IN $KNaO \cdot x\ SiO_2$

Comp. No.	Crystal to Glass Ratio	KNaO to SiO_2 Ratio	Clear Glass Temp. ^{o}C	First Few Crystals	More Crystals	Minimum Pouring Temp.
FF-30	60-40	1 : 3	1200 1100	1040	1010	Below 980
FF-31	60-40	1 : 2	1200 1030	1000	1000	
FF-37	60-40	1 : 1.5	1030	950	900	Below 880
FF-36	60-40	1 : 1	Above 900	900	850 800 750	Below 750
FF-40	40-60	1 : 1	850 800	760	720	Below 700

5. Temperature for Introducing Seeds

In producing crystals it is planned to introduce a seed of PSN into the cooling silicate melt, and to allow further PSN to deposit on the seed. If the temperature at which the seed is introduced is too high, the solution will be insufficiently saturated and the seed will dissolve in the melt. If the temperature is too low, spontaneous crystallization will have started, and the introduced seed will have to compete with the other crystals already present. The seed will then not grow as rapidly as otherwise, and the many crystals present may interfere with each other.

Three preliminary crystals have been grown from seeds, to date. Crystal No. 1 was prepared by placing a seed of PSN in melt FF-30 (see Table IV) at 1100°C, cooling slowly, and removing it at 1040°. Crystal No. 2 was grown by inserting a seed in the same composition at 1040° and quickly dropping the temperature to 980° to 1000°, where it was held for two hours, and then removed. Crystal No. 3 was grown in melt FF-36. The seed was introduced at 990° and withdrawn at 800°. The seeds used, in each instance, were small cubes of pure PSN, about 1 mm on a side, placed in a small basket-like container made of 10-mil platinum wire.

6. Characteristics of Grown Crystals

The crystals grown on the three seeds were annealed on being removed from the molten solution. They were then encapsulated in a clear epoxy (Stycast 1269, cured 16 hours at 200°F). The epoxy mounting served to hold and protect the crystal as it was lapped down far enough to expose a cross section including the original seed. The crystals were then examined with the electron microprobe, with a view to determining the growth mechanisms. The evidence presented indicates that the crystals grew too rapidly. At one stage silicon was not adequately eliminated, and later the growth was so rapid as to surround and trap globules of the solvent glass.

Two factors seem equally responsible for the rapid growth of the crystals: (1) the rate of cooling used in these preliminary experiments is considerably greater than that which

is commonly employed in such crystal growth. It will be advisable now to cool the melts at a slower rate. (2) It is believed that the degree of saturation of PSN is too high in the solutions prepared thus far. There is commonly a significant amount of PSN in the bottom of the platinum crucible during cooling, at all except the highest temperatures and regardless of the rate of cooling (within limits), when the concentration of PSN is of the order of 60 per cent. It is planned to reduce the concentration of PSN to 40 per cent or lower, to lessen the tendency for this precipitation to take place.

Early in the investigation, the composition of the solvent glass was standardized at $KNaO \cdot 3SiO_2$. This is the composition in which crystal No. 1 was grown. Later, other compositions were employed. Glass $KNaO \cdot SiO_2$ currently recommends itself because of its easy solubility in hot water, facilitating the recovery of crystals from the groundmass. However, this glass is readily attacked by water vapor in the air. It may be better, therefore, if possible, to use glass of a compromise composition. It is desired to maintain adequate solubility, but provide somewhat greater chemical durability. The solubility of the $KNaO \cdot 2SiO_2$ glass does not seem to be sufficient for this purpose. It is planned to investigate composition $KNaO \cdot 1.5SiO_2$ to see if it will meet the requirements.

Thus, the work now will be directed at defining more precisely three variables: (1) the rate of cooling the melts, (2) the concentration of PSN to be incorporated into the solutions and (3) the ratio of KNaO to SiO_2 for the molten solvent. Using the newly-defined conditions, seeds will then be introduced at appropriate temperatures and attempts will be made to grow larger, faceted crystals. The information obtained from these crystals will be used in determining the requirements for further refinements and optimization of the growth conditions.

E. RECOMMENDATIONS FOR SPACE EXPERIMENT ON CRYSTAL GROWTH

At this stage of the investigation, the recommendation of the use of a molten glass solvent appears to be well justified. PSN is the favored composition of crystal to be grown, but the basic approach for the development of a space experiment would be

continued if another, similar oxidic composition were selected. Many of the details of a recommended experimental procedure are unresolved, but certain conclusions are beginning to emerge.

The growth of large crystals would be favored by using a large volume of melt, two liters, for instance. However, limitations of space and power call for a small volume of melt and a correspondingly small furnace, particularly for demonstrating feasibility. It is likely, therefore, that 100 ml. of melt, in a furnace occupying 1 to 2 cubic feet, would be a reasonable compromise. The minimum practical volume of melt is assumed to be about 35 ml. (100 grams of material). The 2-liter melt might be appropriate for a scale-up operation.

Power requirements will demand special consideration. Bringing the melt to top temperature prior to starting to grow a crystal should require the largest amount of electrical energy. A simple compact furnace is envisioned, rugged enough to withstand the forces of launch and re-entry. The design maximizes the heat transfer from platinum heater windings to the platinum melting crucible by incorporating the crucible into the system. Heat loss to the surroundings is controlled by a combination of super and fibrous insulation. It is anticipated that the ambient atmosphere for the experiment will be that of the space vehicle. Electrical controls will be consistent with the kind of power available. Depending on the power sources, one will need to plan in terms of using a limited amount of power for a considerable time (slow heating) or of using stored energy at a faster rate for a shorter time (rapid heating). Because slow cooling is so important, an exceptionally well-insulated furnace will be required, and a smaller amount of auxiliary power may still be needed to control the time-temperature schedule during crystal growth.

The need to recover the grown crystals undamaged must be kept in mind. It is hoped that it will continue to look feasible to use hot water as the leaching agent. Fortunately, it appears that adequate standard test procedures have been developed for the physical and electronic evaluation of the crystals. It is at this point, in fact, that the merits of the space preparation of these crystals will ultimately be established.

1. Proposed Program

The proposed program stems directly from the foregoing technical discussions. Problems to be investigated, in essence, include the following:

(a) Final selection of the crystal composition or compositions to be grown
(b) Selection of the composition of solvent glass
(c) Determination of the required concentration of crystal composition in the glass
(d) Determination of the temperature range of crystallization of the selected system
(e) Determination of the configuration and operational characteristics of the furnace
(f) Design and assembly of prototype equipment
(g) Operation of prototype equipment according to proposed schedules and procedures, to assure meeting of program objectives.

The procedures for a typical space experiment in crystal growth are presented in Table VI.

TABLE VI FLOW DIAGRAM OF CRYSTAL GROWTH EXPERIMENT

Initial Preparation (Pre-Launch): (a) Fill crucible with melt, "fine" and solidify (b) Affix seed to sting (c) Lock movable parts (d) Connect and check all wired circuits
Experimental Procedures (In Orbit): (a) Turn on power (settings to be determined) (b) Stabilize melt temperature at _____ °C (to be determined) (c) Unlock furnace hood and rotate to uncover melt (d) Unlock sting dial and insert seed to proper depth (indicator on sting) (e) Release seed and remove sting (instructions will be supplied) (f) Rotate furnace hood to cover position and lock (g) Lower temperature 4°C/hr. for 50 hours (control to be determined) (h) Cut power at °C, cool to RT.
Post-Experimental Procedures: (a) Unlock and remove hood-sting component (b) Disconnect power and thermocouple (c) Unbolt and remove furnace assembly (d) Leach the solidified melt with hot water (e) Recover crystals formed.